Transactions of Illinois Academy of Sciences
(1982), Volume 75, 3 and 4, pp. 283-287

THERMAL HYDROPULPING OF WHEAT STRAW

R. L. Cunningham, R. W. Detroy, and M. E. Carr
Northern Regional Research Center,
Agricultural Research Service,
U.S. Department of Agriculture,*
Peoria, Illinois 61604

ABSTRACT

Thermal hydropulping permits the selective removal and recovery of hemicellulose (pentosans) from wheat straw (WS). WS was pulped with water at predetermined temperatures in the range of 170 to 190°C either 20 or 30 min. After 30 min of thermal hydropulping at 185°C, 94% of the WS pentosans were removed and the residual pulp remained rich in lignin and cellulose. Treatments that disrupt the lignin-hemicellulose-cellulose (LHC) complex prepare biomass for total utilization. The residual pulp and extracted hemicellulose components are promising sources of fermentable sugars for alcohol fuel production.

INTRODUCTION

In 1980 the U.S. harvested nearly 71 million acres of wheat (USDA, 1981) yielding an estimated 100 million tons of residue (Sloneker, 1976). Soil scientists have indicated that nearly half of the residues could be removed from 74% of the Corn Belt acreage without causing a serious soil-erosion problem if farmers used no-tillage conservation practices (Barrett and Hardin, 1982). Previously at our Center, WS was investigated extensively for its potential in board and papermaking (Aronovsky, 1953). The increasing need of American agriculture to become energy self-sufficient has prompted current studies to utilize WS and other biomass as renewable resources for obtaining fuels, chemicals, chemical feedstocks, and feeds. The economics of collection, storage, and transportation of residues will be significant in determining their feasibility for energy and chemicals. Although residues are potentially valuable sources of renewable energy, more efficient pretreatment processes are needed that can destroy fiber structure and disrupt the LHC complex to provide substrates which have greater hydrolytic accessibility (Lipinsky, 1979). Recently, we investigated several pretreatment techniques for disrupting the LHC complex of WS for the primary purpose of substrate bioconversion (Detroy *et al.*, 1980, 1981; Cunningham *et al.*, 1981). We found that an autohydrolysis process (thermal hydropulping) was particularly effective for selectively removing and

*The mention of firm names or trade products does not imply that they are endorsed or recommended by the U.S. Department of Agriculture over other firms or similar products not mentioned.

recovering a hemicellulose (pentosan)-rich fraction from WS. Other workers have reported the effect of various autohydrolysis conditions on the extractability of lignin from hardwoods (Lora and Wayman, 1978) and the separation of lignin, hemicellulose, and cellulose from pine wood with the use of aqueous-organic solvent systems (April *et al.*, 1982).

In our previous report, the pentosan-rich fraction was recovered from the autohydrolyzed WS, converted principally to xylose, and fermented after solvent-extraction to ethanol by *Pachysolen tannophilus* (Detroy *et al.*, 1982b). Concomitantly, enzymatic hydrolysis of the residual cellulose to glucose was substantially enhanced (Cunningham *et al.*, 1981) and provides a substrate for a *Saccharomyces* fermentation (Detroy *et al.*, 1982a). Continued investigation indicates that thermal hydropulping is a promising pulping technique for improving total biomass utilization. We report here the results of process variables which affected the yield, composition, and properties of the WS fractions.

MATERIALS AND METHODS

Soft winter wheat straw (*Triticum* sp., Arthur variety) was chopped in a Taylor, Stiles, and Company chopper and then cut manually to approximately 1-in. lengths. Straw (135 g, moisture-free basis) and water (945 g) were sealed in a 2-liter autoclave and rocked in an electric heating unit. A predetermined temperature in the range of 170 to 190°C was reached in about 40 min and maintained for either 20 or 30 min. The autoclave was removed from the heating unit and cooled with ice until the internal pressure reached zero (generally about 10 min). Subsequently, the contents were discharged into a cloth bag to drain the liquor from the pulp. Residual liquor was removed from the pulp by pressing (Table I) or by washing and pressing (Table II and Fig. 1). Pulp and liquor were placed in a freezer immediately and then freeze-dried before quantitative analyses were performed in duplicate. Crude and alpha cellulose were measured by a monoethanolamine (MEA) method (Nelson and Leming, 1957), lignin by an ultraviolet spectrophotometric technique (Bagby *et al.*, 1973); and xylose in liquor solids by high-pressure liquid chromatography. Alcohol-benzene solubility, ash, and pentosans were determined by TAPPI Official Test Methods.

RESULTS AND DISCUSSION

The effects of pulping temperature on composition and properties of WS cooked with water at 170, 180, and 190°C for 30 min are shown in Table 1. An increase in pulping temperature from 170 to 180°C gave an 8% decrease in pulp yield and an additional 11% loss from 180 to 190°C. Correspondingly, acidity of the liquor increased from pH 5.4 at 170°C to pH 4.9 at 190°C. From 170 to 190°C, major differences in pulp composition are shown by changes in the alcohol-benzene extractives (8 vs. 23%) and pentosans contents (22 vs. 4%). After pulping at 170°C there was no increase in the quantity of alcohol-benzene extractives over that of the original WS. However, at 180 and 190°C there was a 1.7 and 2.3-fold increase, respectively. Alpha cellulose contents of the pulps decreased slightly with increasing pulping temperature above 170°C, which was 2% at 180°C and 11% at 190°C. By increasing the pulping temperature from 170 to 190°C, pentosan removal from the WS was improved from 39 to 90%.

Of the WS lignin, 15, 20, and 43% was removed at 170, 180, and 190°C, respectively. Except for the 190°C cooks, all of the WS lignin can be accounted for in the pulp and liquor solids. When applied to kenaf, a relative standard deviation of 4.3% was reported for this ultraviolet spectrophotometric technique (Bagby *et al.*, 1973). The data viewed at that precision suggest that some decomposition occurred at 190°C. Maximum liquor solids were realized from the 180°C cook. This cook contained also the greatest amount of xylose from hydrolyzed pentosans, which suggests that xylose degradation occurred at 190°C.

The effect of decreasing the cooking time to 20 min is revealed in Table II. These data show little change in pulp yield using the shorter cooking period but reveal a decrease in the extraction of WS pentosans from 79 to 63%. The shorter cooking period changed the alcohol-benzene solubles of the pulp from 1.9-fold over the WS to 1.5-fold and the lignin content from 97 to 62%.

Figure 1 presents pentosans and lignin contents of WS pulps derived from 30 min cooks at 180, 182, and 185°C. Even though there was little change in the lignin contents of these pulps as an effect of temperature, more pentosans were extracted with each increase in temperature, which resulted in a maximum 94% removal of the WS pentosans at 185°C (pulp yield, 66%). These cooking conditions permit selective removal of pentosans as xylans and xylanodextrins and some xylose (Detroy *et al.*, 1982b).

ACKNOWLEDGMENT

We express out appreciation to J. H. Johnson, D. M. Palmer, and W. L. Orton for their analytical and technical assistance.

LITERATURE CITED

April, G. C., R. Bharoocha, J. Sheng, and S. Hansen, 1982. Prehydrolysis Achieves Higher Organosolv Delignification. *Tappi* 65(2):41-44.

Aronovsky, S. I., 1953. Recent Advances in Pulping Straw in the United States. *Tappi* 36(4):167A-171A.

Bagby, M. O., R. L. Cunningham, and R. L. Maloney, 1973. Ultraviolet Spectral Determination of Lignin. *Tappi* 56(4):162-163.

Barrett, J. R., Jr., and B. Hardin, 1982. Corn Cobs for Corn Drying. *Agric. Res.* 30(10):11.

Cunningham, R. L., R. W. Detroy, M. O. Bagby, and F. L. Baker, 1981. Modifications of Wheat Straw to Enhance Cellulose Saccharification by Enzymatic Hydrolysis. *Trans. Ill. State Acad. Sci.* 74(3-4):67-75.

Detroy, R. W., L. A. Lindenfelser, G. St. Julian, Jr., and W. L. Orton, 1980. Saccharification of Wheat-Straw Cellulose by Enzymatic Hydrolysis Following Fermentative and Chemical Pretreatment. Biotech. Bioeng. Symp. 10, John Wiley & Sons, Inc., New York, pp. 135-148.

Detroy, R. W., L. A. Lindenfelser, S. Sommer, and W. L. Orton, 1981. Bioconversion of Wheat Straw to Ethanol: Chemical Modification, Enzymatic Hydrolysis, and Fermentation. *Biotechnol. Bioeng.* 23:1527-1535.

Detroy, R. W., R. L. Cunningham, R. J. Bothast, M. O. Bagby, and A. Herman, 1982a. Bioconversion of Wheat Straw Cellulose/Hemicellulose to Ethanol by *Saccharomyces uvarum* and *Pachysolen tannophilus*. *Biotechnol. Bioeng.* 24: 1105-1113.

Detroy, R. W., R. L. Cunningham, and A. I. Herman, 1982b. Fermentation of Wheat Straw Hemicellulose to Ethanol by *Pachysolen tannophilus*. *Biotech. Bioeng.* (in press).

Lipinsky, E. S., 1979. Perspectives on Preparation of Cellulose for Hydrolysis. *In* Hydrolysis of Cellulose: Mechanisms of Enzymatic and Acid Catalysts. American Chemical Society, Washington, D.C., pp. 1-23.

Lora, J. H. and M. Wagman, 1978. Delignification of Hardwoods by Autohydrolysis and Extraction. *Tappi* 61(6):47-50.

Nelson, G. H. and J. A. Leming, 1957. Evaluation of Monoethanolamine Method of Cellulose Determination for Agricultural Residues. *Tappi* 40(10):846-850.

Sloneker, J. H., 1976. Agricultural Residues, Including Feedlot Wastes. Biotech. Bioeng. Symp. 6, John Wiley & Sons, Inc., New York, p. 238.

U. S. Department of Agriculture, 1981. *In* Agricultural Statistics, U.S. Government Printing Office, Washington, D.C., p. 433.

I. Composition and properties of thermal hydropulped wheat straw[a]

Analyses, %	Pulping temperature, °C			
	unpulped	170	180	190
	Wheat straw	Pulp		
Yield[b]	——	79.1	73.0	65.3
Solubility in alcohol-benzene	6.5	8.2	15.2	22.9
Cellulose				
MEA[c]	48.2	54.6	52.4	52.1
Alpha	30.2	38.3	40.4	41.4
Pentosans	29.1	22.5	13.9	4.4
Lignin	14.5	15.5	15.9	12.5
Ash	9.5	7.6	9.4	8.8
		Liquor solids		
pH[d]		5.4	5.2	4.9
Yield[b]		17.1	21.3	14.8
Lignin		14.0	13.5	28.6
Xylose		0.0	5.5	3.8

[a]Water:straw (1-in. lengths) = 7:1; 30 min at cooking temperature following a 40-min heat-up.
[b]Original wheat straw, moisture-free basis.
[c]Monoethanolamine method (Nelson and Leming, 1957).
[d]pH of liquor after pulp pressing.

II. Effect of pulping time on composition and properties of wheat straw pulp[a]

Analyses %	Pulping time, min	
	20	30
Yield[b]	73.4	72.0
Liquor pH[c]	3.8	4.0
Solubility in alcohol-benzene	13.1	17.0
Cellulose		
MEA[d]	53.7	52.2
Alpha	42.2	40.6
Pentosans	14.7	8.5
Lignin	12.3	19.6
Ash	8.2	8.8

[a]Water:straw (1-in. lengths) = 7.1, 180°C cooking temperature following a 40-min heat-up.
[b]Original wheat straw, moisture-free basis.
[c]pH of liquor at end of cook.
[d]Monoethanolamine method (Nelson and Leming, 1957).

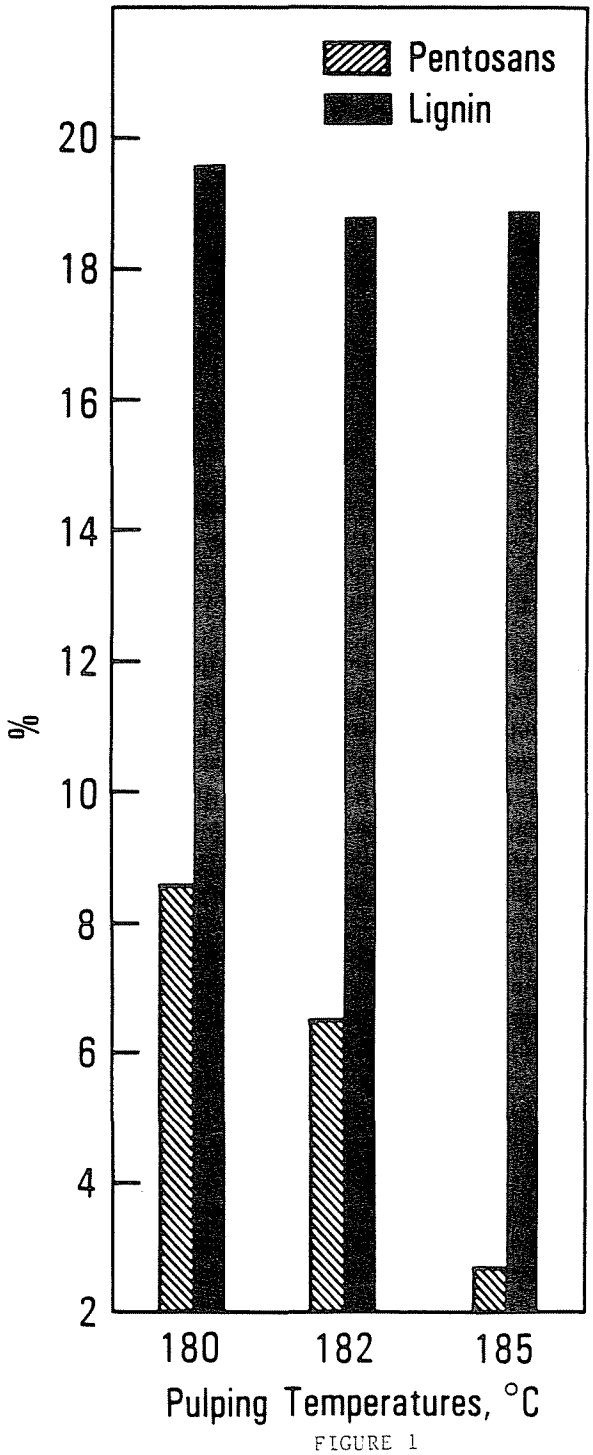

FIGURE 1

FIGURE LEGEND

1. Lignin and pentosan contents of wheat straw pulps as a function of cooking temperature. Water:straw (1-in. lengths) = 7:1. Thirty-min cook.